

German Patent No. 2 048 721 (Offenlegungsschrift)

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Translated from German by the Ralph McElroy Co., Custom Division  
P. O. Box 4828, Austin, Texas 78765 USA

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## PATENT OFFICE

PATENT NO. 2 048 721

(Offenlegungsschrift)

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## DISPOSABLE ABSORPTIVE ARTICLE

Applicant:	Union Carbide Corporation, New York, NY (USA)
Inventor:	Per Gunnar Assarsson Montclair, NJ (USA)
Agent:	H. Görtz, patent attorney 6000 Frankfurt

Search application according to § 28a of the Patent Law has been made.

The invention pertains to absorptive articles that are disposable after use. It especially pertains to such articles that contain a stabilized hydrophilic polyethylene oxide that is soluble in water. Articles that can be considered as such are: diapers, sanitary napkins, sanitary cloths, dental rolls, surgical sponges, cloth for baby deliveries, nonconnected bolsters, and the like. The invention additionally pertains to stabilized insoluble hydrophilic polyethylene oxides.

Absorptive articles for absorbing body fluids that are disposable after use have been used for a long time. However, they are not always satisfactory. Many materials, formulations, and absorbing agents are described in the literature. Many such articles that are presently on the market have the disadvantage that they absorb little or poorly. Heretofore, in order to improve the absorptive capacity, among other things, cheap materials that have good absorptive properties have been added, such as fluffy wood cellulose and the like. These materials were arranged between layers of liquid-permeable textiles. Although they absorbed liquids to a satisfactory extent, in many cases these articles were too bulky and not comfortable. For example, diapers must have a minimum thickness in order to take up sufficient amounts of body fluids. However, if the material has a low absorptive capacity, the diaper has to be very thick and therefore can only poorly adjust to the body.

An additional great disadvantage of conventional articles consists of the fact that although they can absorb large amounts

of liquid, a part of the absorbed liquid can be squeezed out with pressure. This is due to the fact that the liquid is physically enclosed in a fibrous structure, whereby even a relatively low pressure suffices to squeeze the fluid out of the absorbing agent. This is naturally undesirable, especially in sanitary napkins.

U.S. Patent No. 2,849,000 describes an improved sanitary napkin in which the absorptive cotton fiber is coated with an agent that increases the absorptive capacity. Typical agents are, for example, sorbitol, mannitol, glycerin, glycols, and polyglycols. The substances are applied by spraying onto the cotton or in washing with liquids. Although such substances have a good absorptive capacity, they are soluble and can therefore be extracted from the cotton fibers by body fluids.

Recently, it has been found that insoluble hydrophilic polyethylene oxides are especially useful in the preparation of absorptive articles that are disposable after use. They absorb relatively large amounts of liquid and retain it even under pressure. Although these polymers are stable, even for longer periods of time in the presence of oxygen at temperatures of up to 25°C, they are nevertheless degraded at higher temperatures of approximately 40°C in the presence of even small amounts of oxygen. Thus, for example, an unstabilized polymer after 6 weeks of storage at 40°C in water containing only dissolved oxygen is 100% soluble in water. The water solubility of an initially insoluble polymer is a useful indicator of the degree of degradation.

The water-soluble polyethylene oxides, which are the semi-finished products in the preparation of water-insoluble

hydrophilic polyethylene oxides also have the disadvantage that they are degraded upon oxidation. Many stabilizers for soluble polymers have already been proposed. In spite of the structural similarity between the soluble polymers and the insoluble polymers, no relation has been found from which the stabilizing capacity for insoluble polymers could be ascertained from the stabilizing capacity for soluble polymers. Thus, for example, certain alcohols such as isopropanol or propylene glycol can be used to stabilize soluble polymers. However, these alcohols are inadequate or completely ineffective in stabilizing insoluble polymers.

One objective of the invention involves absorptive articles that are disposable after use and do not have the above-indicated disadvantages at all, or do so to only a limited extent. An additional objective of the invention involves improved absorptive articles that are disposable after use and contain a stabilized insoluble hydrophilic polyethylene oxide as an absorbing agent. A further objective of the invention involves such articles with an improved absorptive capacity.

A still further objective of the invention involves absorptive articles that contain a stabilized insoluble hydrophilic polymer, which is a crosslinked polyalkylene oxide, especially a crosslinked polyethylene oxide. A further additional objective of the invention involves stabilized crosslinked polyethylene oxides. One more objective of the invention involves absorptive articles that are disposable after use, in which the polymer is practically insoluble in body fluids. These and additional objectives of the invention will be obvious to those skilled in the art from the description.

The invention pertains to absorptive articles that are disposable after use, which contain a water-insoluble hydrophilic polyethylene oxide that is stabilized with a polyethyleneimine.

Polyethylene oxides that have been rendered insoluble in water by crosslinking are especially valuable because they can take up large amounts of liquid, e.g., of water, at about 25-fold to 100-fold their dry weight. These polymers not only can take up water in large amounts, but they are insoluble in water independently of the temperature, can retain liquids, solutions, and suspensions, and are gels.

The nonstabilized hydrophilic polyethylene oxides that are insoluble in water are well known. Their characteristics and preparation are described, for example, in U.S. Patent No. 3,264,202. The insoluble polymers are obtained by the treatment of water-soluble polyethylene oxides with ionizing radiation of such strength that crosslinking occurs and water-insoluble polymers are formed. The water-soluble polyethylene oxides that can be used as starting materials are likewise well known and are described, for example, in U.S. Patent Nos. 3,127,371, 3,214,387, 3,275,998, 3,398,199, and 3,399,149.

The water-soluble polyethylene oxides are homopolymers of ethylene oxide or copolymers of ethylene oxide with one or several other alkylene oxides such as propylene oxide, 1,2-butylene oxide, 2,3-butylene oxide, isobutylene oxide, styrene oxide, and the like. These copolymers contain residues of ethylene oxide in such amounts that the copolymer is water soluble. The minimum amount of ethylene oxide residue varies somewhat as a function of the type of comonomer used. Generally, the copolymers contain at least approximately 50 wt% of a residue

of ethylene oxide, preferably at least 75 wt%. The homopolymer of ethylene oxide is preferred.

The water-soluble polyethylene oxides generally have a molecular weight such that the reduced viscosity of the polymer is approximately 0.5-75 and above, preferably approximately 1-60. Aqueous solutions or dispersions at 25°C at a concentration of 5 wt% have a viscosity of approximately 225 centipoise, wherein the viscosity of 1% solutions of the same can increase to approximately 12,000 centipoise.

The reduced viscosity is measured as follows: 100 mL of acetonitrile are placed into a round-bottomed flask with approximately a 250-mL capacity and containing a screw cap. With constant stirring, 0.200 g of the polymer is added to the flask, wherein the weighing is done at an accuracy of  $\pm 1$  mg. The screw-cap of the flask is then lined with a piece of aluminum foil, carefully applied to the flask, and screwed tightly. Subsequently, the flask is placed in a suitable rolling [sic; gentle mixing] arrangement with a roller having a 15-cm diameter and is rolled for  $16 \pm 0.5$  h. After removal from the roll, the solution is passed under pressure through a coarse filter of sintered glass. Thereafter, the time, in seconds, that a sample of the solution requires to pass through a calibrated Ubbelohde viscometer at a temperature of  $30 \pm 0.01^\circ\text{C}$  is determined. For this, a stopwatch whose scale is divided into 0.1-sec units and which during the course of 60 min does not deviate from the exact time by more than 0.1% is used.

The time, in seconds, that the acetonitrile solution requires to pass through the viscometer is measured. A calculation is then done using the following formulas:

$$AS - F/AS = AC$$

$$SS - F/SS = SC$$

$$(SC - AC)/AC = SV$$

$$SV/K = RV$$

Here, the individual symbols have the following meaning:

F = the correction for the viscometer

AS = the [number of] seconds needed for the acetonitrile

AC = the corrected seconds [sic] needed for the acetonitrile

SS = the seconds needed for the polymer solution

SC = the corrected seconds needed for the [polymer] solution

SV = specific viscosity

RV = reduced viscosity

K = concentration in grams per 100 mL of acetonitrile

For example, the table below shows the relation between the average molecular weight of the polyethylene oxide, the reduced viscosity, and the viscosity in water.

Wt% polymer in acrylonitrile	Reduced viscosity	Average molecular weight	Viscosity of aqueous solution at 25°C
0.2	1.5	150,000	200 cps (5% soln.)
0.2	60	10,000,000	7000-9000 cps (1% soln.)

The viscosity in water is the viscosity of a solution of a given concentration of the polymer in water, measured with a Brookfield viscometer Model RVF using a No. 1 spindle at



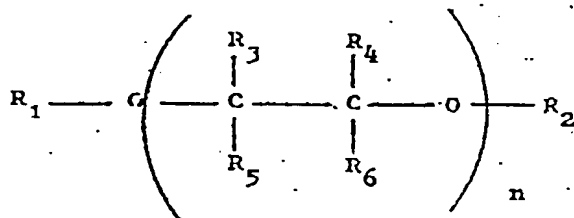
2 rev/min. The viscosity is measured at room temperature, i.e., at approximately 24°C.

The water-insoluble hydrophilic polyethylene oxides are produced by treating the above-described water-soluble polyethylene oxides with sufficiently strong ionizing radiation to obtain a reaction and insolubilization of the polymer, wherein a water-insoluble hydrophilic polymer is formed. The expression "ionizing radiation" refers to such radiation as is sufficient to attain electronic [sic; electron] stimulation and/or ionization in the molecules of the polymers when a solvent is used [sic; states "solid state" below], but is not strong enough to affect the nucleus of the atom. Conventional sources for suitable ionizing radiation are gamma rays produced by radioactive isotopes such as  $\text{Co}^{60}$  and  $\text{Cs}^{137}$ , spent nuclear-fuel rods, conventional X-rays and electrodes from van de Graaf accelerators, linear electronic [sic] accelerators, resonance transformers, and the like. The suitable ionizing radiation for the purpose of the invention has an energy level of approximately 0.05-20 MeV.

The irradiation of the noncrosslinked water-soluble polymer can be done in the solid state or in solution. Solid polymers can be irradiated in air, in vacuum, or in various other gases. Irradiation in solution can be done in a solution of the polymer in water or in a mixture of water and a miscible organic solvent. Any desired conventional process can be used to bring the solid polymer or a solution thereof into contact with the ionizing radiation. Such processes are well known to those skilled in the art.

The amount of ionizing radiation to which the polymer is exposed depends on various parameters. If the irradiation is done at low power and in the presence of a rinsing gas such as oxygen for free radicals, very high total doses are required to produce the water-insoluble hydrophilic polymer. However, if the irradiation is done under such conditions that the free radicals formed have a relatively long life, i.e., if the irradiation with high power is done in the absence of oxygen or in a solution where the oxygen is quickly consumed, then water-insoluble hydrophilic polymers are very easily formed. The preferred process for preparing the water-insoluble hydrophilic polymers is irradiation in an aqueous solution of the water-soluble polymer, wherein ionizing radiation with a strength of approximately 0.10-20 MeV is used, with a total dose of approximately 0.05 to 10 Megard [sic; megarad].

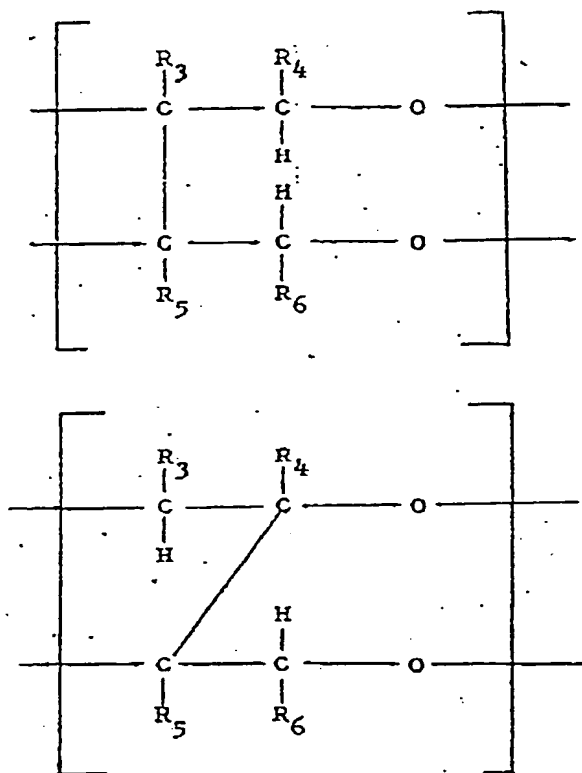
The polymeric hydrophilic gels have the general formula:



wherein they are crosslinked.  $R_1$  and  $R_2$  are the same or different and represent a hydrogen atom, an alkyl residue, or an alkyl-substituted aryl residue.  $R_3$  to  $R_6$  are the same or different and

represent a hydrogen atom, a methyl residue, a phenyl residue, or a vinyl residue;  $n$  is greater than 1.

These hydrophilic gels contain at least one of the following structural units:

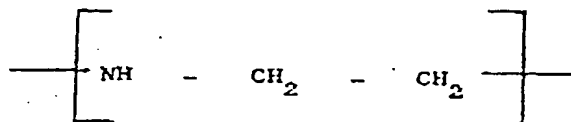


Here  $R_3$  to  $R_6$  have the above-indicated meaning.

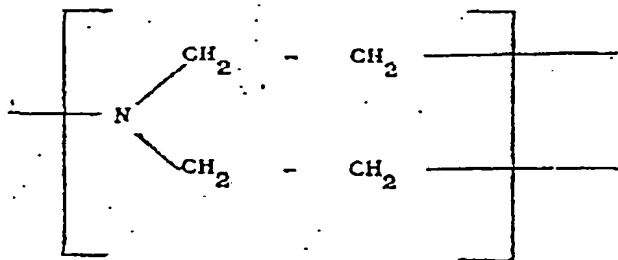
The designation "insoluble" or "making insoluble" designates the formation of a polymer of which a variable proportion is

practically insoluble in water, depending on the radiation dose. These polymers can swell and take up many times their weight of water because they are also hydrophilic.

As already indicated, the articles of the invention contain water-insoluble hydrophilic polyethylene oxides that are stabilized with a polyethyleneimine. Polyethyleneimines are polymers of ethyleneimine and primarily contain the following [repeating] units:



and a small proportion of the following [repeating] units:



Generally, the polyethyleneimines have molecular weights of approximately 10,000-100,000 and are thereby well-suited for the application of the invention. Especially preferred are polyethyleneimines with molecular weights of approximately

20,000-80,000. Such polymers can be prepared according to well-known processes and are commercially available.

In performing the process, the polyethyleneimines are used in such amounts that they stabilize [the polyethylene oxides]. The expression "stabilizing amount" refers to that amount of polyethyleneimine that, after the addition of polyethylene oxide, lowers the degradation rate compared to the degradation rate of the unstabilized polymer. Approximately 0.01 wt%, based on the solid polyethylene oxide, suffices to stabilize the polymer against degradation. For most [cases], one preferably uses the stabilizer in amounts of approximately 0.2-3.0 wt%. However, if desired, the polyethyleneimine can be used in an amount of up to 50 wt%.

The stabilization of the water-insoluble hydrophilic polyethylene oxide can be done in the various processing steps. For example, one can add the polyethyleneimine to an aqueous solution of polyethylene oxide, cast a film from this solution, and subsequently crosslink. The addition of the stabilizer to the solution of polyethylene oxide also stabilizes the solution itself. However, one can also mix the pure solid polymer with the stabilizer, extract the mixture, or calender it to produce a film or some other form. It is then crosslinked by irradiating the film or the shaped object. By adding the stabilizer before extruding or calendaring, the stability of the polymer melt is also increased at increased temperature. Finally, the water-insoluble hydrophilic polyethylene oxide can be stabilized after irradiation, specifically by absorption of the stabilizer from a solution.

The stabilized hydrophilic polyethylene oxide can easily be worked into well-known or commercial articles in which special weight is given to a good absorptive capacity. For example, the polymers can be worked into diapers of the type that are described in U.S. Patent Nos. 2,788,003, 2,849,000, 2,860,637, 3,306,293, and 2,667,168. One can work the polymers into tampons or sanitary cloths such as are described in U.S. Patent Nos. 3,121,427 and 3,070,095. The polymers can be used in various ways, e.g., as a powder that is bound and dispersed in a substrate of cellulose or a similar material, or as a film of the crosslinked polymers, which lies in a layer between the support layers. Here, well-known processes can be used to bind the film or the powder to the support.

The amount of hydrophilic gel used depends on the type of absorptive article and its intended use. Absorptive articles can contain approximately 2-98 wt% of the gel.

The following examples explain the effectiveness of the stabilizers of the invention to improve the stability of the insoluble hydrophilic polyethylene oxide.

For Examples 1 to 3, the insoluble hydrophilic polyethylene oxide was prepared in such a manner that a 4% solution of the homopolymer of polyethylene oxide, with a reduced viscosity of approximately 60, was irradiated using 1,000,000 electron volts with a total dose of approximately 1 megarad. The ionizing radiation was produced in a van de Graaf accelerator.

The polymer was prepared as a 7.5-cm-wide strip with a weight of approximately 3 g/m. It had [sic; was applied to] a thin, open support with a gauze-like structure. The strips were

not dried and were treated in gel form according to the following standard procedure.

Two pieces of the above-described strips of approximately 30-cm length were placed in a vessel. In the vessel, there was a solution of various amounts [sic] of polyethyleneimine in 500 mL of distilled water. The vessel was loosely covered and placed in an air oven at 40°C. Periodically, the rigidity of the polymer was determined by testing the stiffness of the strip. Likewise, the pH value of the solution was periodically measured because degradation is generally accompanied by a lowering of the pH value. The findings were characterized by the symbols given below:

- (+) the strip is solid, no notable degradation
- (±) the strip is still coherent, but notable degradation can be determined by the softness or stickiness of the surface
- (-) the strip is 100% destroyed and 100% soluble in water.

The stabilizers used, their concentration, and the results are described in the following examples.

#### Example 1

In this example, a strip was prepared in the above-described manner, but without using polyethyleneimine as a stabilizer. This strip was used as a comparison. In the above-described standard test, the following results were obtained:

Table I.

Time duration	Appearance of strip	pH value
3/4 month	(-)	3.5

Example 2

0.215 g of polyethyleneimine with a molecular weight of approximately 50,000 was dissolved in 500 mL of water, whereby a  $10^{-3}$ M solution of polyethyleneimine monomer units [sic] was formed. The strips were introduced into this solution. The following results were obtained:

Table II.

Time duration	Appearance of strip	pH value
3/4 month	(+)	9.4
1 3/4 months	(+)	8.9
3 3/4 months	(+)	8.4
6 months	(+)	8.8

Example 3

0.0002 g of polyethyleneimine with a molecular weight of approximately 50,000 was dissolved in 500 mL of water, whereby a



$10^{-5}$ M solution was formed. The strips were introduced into this solution. The following results were obtained.

Table III.

Time duration	Appearance of strip	pH value
3/4 month	(+)	7.4
1 3/4 months	(+)	7.4
3 3/4 months	(+)	7.3
6 months	(+)	8.4

#### Example 4

This example shows the stabilization of a hydrophilic polyethylene oxide by the addition of the stabilizer to an aqueous solution of the polyethylene oxide before crosslinking. The polyethyleneimine stabilizes the solution of the polyethylene oxide before and after crosslinking.

Three aqueous solutions were prepared, which were comprised as follows: (A) into a 4% solution of the homopolymer of polyethylene oxide with a reduced viscosity of approximately 60, 0.13 wt% of the homopolymer of polyethyleneimine with a molecular weight of approximately 50,000 was added in such a manner that it contained monomers in a molecular ratio of 30:1. (B) The procedure of (A) was followed, with the difference that 0.04 wt% of polyethyleneimine was added so that the solution contained these two components in a molar ratio of 100:1. (C) The procedure

of (A) was used, with the difference that 0.027 wt% of polyethyleneimine was added so that the solution contained the two components in a molar ratio of 150:1.

These solutions were crosslinked with ionizing radiation with an energy of 1,000,000 electron volts, and a total dose of approximately 1 megarad was used. The ionizing radiation was produced by a van de Graaf accelerator. The polymer gels produced by this were strips with a width of approximately 7.5 cm and had a weight of approximately 50 g/m. These strips were laid upon an open support with a gauze structure. The strip was dried in air at room temperature for approximately 16 h to form a film.

In comparative tests without the addition of polyethyleneimine, the strip was prepared in a corresponding manner and dried.

This example shows the effectiveness of the added polyethyleneimine in three different amounts in order to stabilize the dried hydrophilic insoluble polyethylene oxide.

15-cm-long sections of the stabilized and nonstabilized strips were enclosed in a 19 x 7.5 cm strip of cotton, rolled together, and placed in an air oven at 90°C. After various [heating] times, the rolls were removed, whereupon the strips were placed in a solution of 500 mL of methanol containing 10 wt% of water in order to extract the degraded soluble polymer. Table IV shows the weight of the strip without the gauze immediately after drying, the weight of the strip after treatment in the air oven and the subsequent extraction, and the proportion of polymer degraded in this manner.

Table IV

Sample	Time in oven, h	Weight in g		Degradation %
		Before treatment	After in oven	
Nonstabilized polyethylene oxide				
1	1	0.7735	0.7064	17.9
2	1.5	0.9574	0.7702	32.7
3	2.5	0.8919	0.6678	46.1
4	3	0.9448	0.6037	63.6
5	4	0.6891	0	100
Stabilized polyethylene oxide with solution (A)				
6	1.5	0.6540	0.6195	5.3
7	4	0.6815	0.5610	17.7
8	5	0.4976	0.4174	16.1
9	8.5	0.7428	0.6505	12.4
10	23.5	0.7195	0.5441	23.4
Stabilized polyethylene oxide with solution (B)				
11	3	0.7380	0.7321	0.8
12	6	0.6890	0.5780	16.8
13	25	0.6855	0.4526	23.8
Stabilized polyethylene oxide with solution (C)				
14	3	0.5228	0.5184	0.8
15	6	0.5346	0.3587	32.9
16	25	0.4480	0.1047	76.6

As already indicated, the polyethyleneimine stabilizes the solution of polyethylene oxide before crosslinking. This was shown by the preparation of a 2% solution of polyethylene oxide with a concentration of 2.5 wt% of polyethyleneimine, based on the weight of the solid polyethylene oxide. This solution and a 12% solution of polyethylene oxide without a stabilizer, serving

as a comparison, were held for various times at 40°C. The viscosities were measured in centipoise in a Brookfield viscometer and are given in Table V. The decrease in the viscosity is an indication of the degradation of the molecular weight.

Table V. Viscosity of solutions of polyethylene oxide in centipoise.

Sample	Time in days		5	22	78	106
	1	2				
Comparison sample	54,000	40,000	50	-	-	-
Stabilized solution	56,000	52,000	57,000	61,000	40,000	28,000

#### Example 5

A different process for the preparation of films of hydrophilic polyethylene oxide consists of using a melt of solid polyethylene oxide that is [then] mixed, extruded, and calendered. The film formed in this way is crosslinked by irradiation. It is well known that degradation occurs during melting, mixing, and shearing if the temperature is increased. This example shows the stabilizing effect of the addition of polyethyleneimine to the solid polyethylene oxide. Brabender curves were obtained with solid polyethylene oxide having a

molecular weight of approximately 600,000, whereby No. 6 rollers with a jacket temperature of 150°C were used. 26 g were processed in each case.

The head of the mixing vessel was rapidly fed at 40 rpm and the instrument was read on the 5X scale. Three minutes later, the scale was set at 1X [not clear] with a preloading of 500 m-gm [sic]. At the same time, the rate was increased to 100 rpm. The curve was recorded until the torque had decreased to 75% of its initial value. The time required to reach this point serves as a measure of the stabilizing effect of the stabilizer. This time was measured from the point at which the rate of rotation was increased to 100 rpm. The initial torque was also read at this point. The results are given in Table VI.

Table VI.

Sample No.	% Stabilizer	Initial torque $T_0$ , m-gm	Time to attain 75% of initial torque, in min
1	0	1280	4.5
2	1.6	1380	23.0
3	0.25	1380	8.5
4	0.12	1380	7.5

The rate of decrease of the torque was also calculated from the slope of the curve at which 75% of the initial torque was attained. The results are given in Table VII.

Table VII.

Sample No.	% Stabilizer	Rate of decrease of the torque m-gm/min
1	0	88.9
2	1.6	9.1
3	0.25	44.5
4	0.12	48.8

These figures show that an addition of 0.25% of polyethyleneimine decreased the rate of decrease of the torque by half, compared to unstabilized polyethylene oxide.

In Sample 1, the temperature increased after approximately 7.5 min, measured from the start of the experiment, to a maximum of 169°C, then decreased very rapidly while the degradation after 15 to 16 min attained an equilibrium with the temperature [sic]. Sample 2 reached a maximum temperature of 175°C after approximately 14 min, which decreased only by approximately 1°C in the next few minutes of the process. Sample 3 reached a maximum temperature of 174°C after approximately 9 min, whereupon the temperature gradually decreased. Sample 4 behaved approximately like Sample 3.

#### Example 6

Tampons were produced in the shape of approximately 1 m-long and 7.5-cm-wide strips that consisted of two pieces of dried polymer as in Example 4, and were laid upon a thin gauze support. This support [sic; strip] was produced by the crosslinking of

polyethylene oxide with polyethyleneimine in solution at a monomer ratio of 30:1. The polymer, at a weight of 5.1 g/m, was arranged between three pieces of a cotton textile at 13 g/m. The thus-produced objects were two-layered tampons. In a similar manner, tampons were prepared with only one piece of the polymer, which was arranged between three pieces of the same cotton sample. These tampons are designated as single-layered. For comparison, additional tampons were produced, consisting of three pieces of the same cotton sample with a weight of 13 g/m.

The absorption capacity of these different tampons was measured in three different arrangements, whereby, as absorbed liquids, water, a physiological aqueous saline solution, and blood treated with citrate solution, were used.

(1) Arrangement I contained an approximately 12.5-cm-tall bottle with a diameter of approximately 7.5 cm. The bottle was filled with a 10% solution of polyethylene. A glass rod with a diameter of 10 mm and a length of 11.5 cm was arranged in the middle. The bottle and contents were illuminated [sic; irradiated] with  $\text{Co}^{60}$  at a total dose of approximately 1 megarad. After removal of the rod, the gel obtained and the container were tilted mouth-downward at an angle of  $40^\circ$ . Connected by a plastic tube with a burette of 50 mL [sic; incomplete sentence]. The plastic tube was passed through the back of the gel [sic] to the middle hole. The tampon to be tested was introduced in its entirety through the opening of the hole. The liquid, in this case water, was introduced from the burette in a regulated amount of approximately 10 to 20 mL/h into the hole. After saturation of the tampon at the respective pressure in the arrangement, the

liquid passing through was collected and measured. The tampon was weighed before use and in the saturated state after the end of the experiment. Arrangement II was similarly constructed, with the difference that the glass rod had a diameter of 7 mm. Otherwise, the procedure was as for Arrangement I. As the liquid, a physiological aqueous saline solution was used which was introduced from the burette into the hole at a regulated rate of 10 to 22 mL/h. As in Arrangement I, the liquid passing through was collected and measured, and the tampon was weighed before and in the saturated state after the experiment.

The absorption capacities of the single-layered tampon (hydrogel and gauze), the two-layered tampon, (hydrogel and gauze), and the comparison tampon are given in Table VIII.

Table VIII. Absorption testing of tampons with stabilized hydrophilic polyethylene oxide<sup>(1)</sup>.

Sample	Water uptake	Dry weight of the tampon	Uptake capacity g of liquid per g of tampon	Increase in uptake capacity compared to comparison sample
Comp. sample of conv. tampon	11.6 g	3.0 g	3.90	-
Single-layered film with gauze	13.3 g	3.1 g	4.30	10.3



Two-layered film with gauze	17.1 g	3.3 g	5.20	33.3
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(1) In vitro at a pressure of 0.035 kg/cm<sup>2</sup>. 30:1 molar ratio of the monomers.

The absorptive capacity of test tampons of two-layered films without gauze, of two-layered films of the hydrogel with gauze, and comparison tampons are given in Table IX.

Table IX. Absorptive capacity of tampons with stabilized hydrophilic polyethylene oxide<sup>(1)</sup>.

Sample	Saline uptake	Dry weight of the tampon	Uptake capacity g of liquid per g of tampon	Increase in uptake capacity compared to comparison sample%
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Comp. sample of conv. tampon	10.25 g	3.0 g	3.42	-
Two-layered film with gauze	14.3 g	3.25 g	4.40	29.4
Two-layered film w/o gauze	16.3 g	3.25 g	5.02	47.0
Blood treated with citric acid				

Two-layered  
film with  
gauze        16.2 g

- (1) As in Table VIII, with the exception that the pressure was 0.07 kg/cm<sup>2</sup>.

#### Example 7

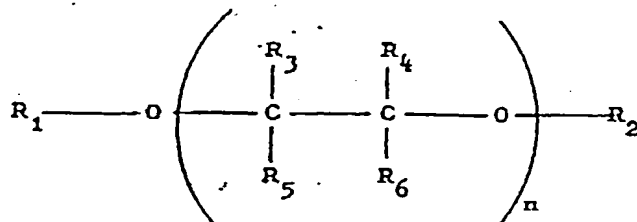
Besides being used to produce tampons, the hydrogels of the invention can also be used for sanitary cloths. A test sample was prepared in the following manner.

A 10% solution of high-molecular-weight polyethylene oxide was poured into the open end of a round polyethylene mold that had a diameter of 6.4 cm and a length of 12.5 cm. In the middle of the mold, there was a glass rod with a diameter of 9.5 mm that extended from the back into the mold at a depth of approximately 13 mm. The entire [preparation] was irradiated with Co<sup>60</sup> at a total dose of approximately 1 megarad, wherein a coherent but flexible gel was formed. After removal of the glass tube, a burette was attached to the back of the mold at the middle of the hole. From this, a test liquid, water or saline, was introduced into the hole at a regulated rate. The mold was inclined at an angle of approximately 45°, wherein it was attached with a clamp. The mouth of the hole pointed downwards. The mold was equipped with a sanitary cloth. Commercial cloths with the trade name "Kotex" were arranged in layers between two layers of polyethylene oxide and polyethyleneimine [sic; misleading] that were produced as in Example 4 (A). The cloths were applied to the

mold, wherein approximately 1 mL/min was introduced. The cloths produced according to the invention had a significantly better absorptive capacity because of the crosslinked polymer than did the comparison cloths.

### Patent claims

1. Disposable absorptive article, characterized by the fact that it contains a hydrophilic crosslinked polymer of the general formula



wherein  $R_1$  and  $R_2$  are the same or different and represent a hydrogen atom, an alkyl residue, or an alkyl-substituted aryl residue,  $R_3$  to  $R_6$  are the same or different and represent a hydrogen atom, a methyl residue, a phenyl residue, or a vinyl residue, wherein  $n$  is greater than 1, and whereby the polymers contains at least the amount of polyethyleneimine required for stabilization.

2. Article according to Claim 1, characterized by the fact that the hydrophilic polymer is a crosslinked polyalkylene oxide.

3. Article according to Claim 2, characterized by the fact that the hydrophilic polymer is a crosslinked polyethylene oxide.

4. Article according to Claims 1 to 3, characterized by the fact that it is a sanitary napkin.

5. Article according to Claims 1 to 3, characterized by the fact that it is a sanitary cloth.

6. Article according to Claims 1 to 3, characterized by the fact that it is a tampon.

7. Article according to Claims 1 to 3, characterized by the fact that it is a diaper.

8. Process for stabilizing a water-insoluble hydrophilic polyethylene oxide, characterized by the fact that at least the amount of polyethyleneimine required for stabilization is added and for subsequent crosslinking.

9. Process for stabilizing a water-soluble polyethylene oxide, characterized by the fact that at least the amount of polyethyleneimine required for stabilization is added.